contracted H-atom scattering factor from Stewart, Davidson & Simpson (1965).

Unit-cell dimensions and intensity data: Siemens diffractometer software (Siemens, 1990). Diffraction data reduction and error analysis: local programs (Blessing, 1989). Structure determination: *DIRDIF* (Beurskens *et al.*, 1992) as implemented in *TEXSAN* (Molecular Structure Corporation, 1992). Least-squares refinement: *SHELXL*93 (Sheldrick, 1993). Structure drawings: *ORTEP*II (Johnson, 1976); *PLUTO* (Motherwell, 1979) as implemented in *TEXSAN* and *PLATON* (Spek, 1990).

We are grateful for support of this research by Fulbright Fellowships to WM and WK, grant No. 3 03 02 91 01 from the Polish Committee for Scientific Research to WK, and NIH grant No. GM34073 to RHB.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1167). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Blessing, R. H. (1989). *J. Appl. Cryst.* **22**, 396–397, and references cited therein.

Brock, C. P., Schweizer, W. B. & Dunitz, J. D. (1991). *J. Am. Chem. Soc.* **113**, 9811–9820.

Cole, F. E. (1968). *Am. Crystallogr. Assoc. Meet.*, Tucson, Arizona, February. Abstract No. F8, p. 35.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, edited by W. C. Hamilton & J. A. Ibers, p. 71. Birmingham: Kynoch Press.

Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.

Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*, pp. 40–42. Berlin: Springer-Verlag.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kwiatkowski, W., Maniukiewicz, W. & Blessing, R. H. (1994). *Am. Crystallogr. Assoc. Meet.*, Atlanta, Georgia, June 1994. Abstract No. PBO-05, p. 95. *IUCr Sagamore XI Conference on Charge, Spin, and Momentum Densities*, Brest, France, August 1994, Abstract No. P4–58. In preparation.

Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Programs*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, S. (1979). *PLUTO. Program for Plotting Molecular and Crystal Structures*. University of Cambridge, England.

Putkey, E. & Sundaralingam, M. (1970). *Acta Cryst.* B**26**, 782–789.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1990). *Diffractometer Control Software*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Slone, C. J. & Cole, F. E. (1971). *Am. Crystallogr. Assoc. Meet.*, Ames, Iowa, August 1971. Abstract No. G9, p. 56.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Sundaralingam, M. & Putkey, E. F. (1970). *Acta Cryst.* B**26**, 790–800.

Wallach, O. (1895). *Liebigs Ann. Chem.* **286**, 90–143.

Acta Cryst. (1996). C**52**, 1741–1743

3-Isopropylamino-4-methyl-4*H*-pyrido-[4,3-*e*][1,2,4]thiadiazine 1,1-Dioxide

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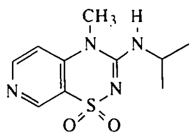
(*Received 16 January 1996; accepted 8 February 1996*)

Abstract

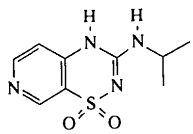
The title compound, $C_{10}H_{14}N_4O_2S$, is the 4-methyl analogue of an original potassium channel opener molecule related to diazoxide. The crystal structure determination of a compound with a methyl substituent in the 4-position provides geometric reference data which may be useful for analysing the preferential 2*H*- or 4*H*-tautomeric form adopted in unsubstituted derivatives of this class of compounds in the solid state.

Comment

The title compound, (I), is the methyl analogue of (II), a potassium channel opener structurally related to diazoxide [7-chloro-3-methyl-2*H*(or 4*H*)-1,2,4-benzothiadiazine 1,1-dioxide] (Bandoli & Nicolini, 1977; Pirotte *et al.*, 1993; de Tullio *et al.*, 1996).



(I)



(II)

The present crystallographic investigation of (I) will help our knowledge of the conformational behaviour of 4-methyl-substituted derivatives compared with unsubstituted derivatives of this class of heterocyclic compounds. Indeed, the presence of a methyl substituent in the 4-position of the thiadiazine ring imposes the adoption of a 4*H*-tautomeric form for (I). Thus, comparison of the N2—C3 [1.326 (4) in (I) and 1.315 (4) Å in (II)] and C3—N4 [1.381 (4) in (I) and 1.366 (4) Å in (II)] bond lengths of the two compounds may allow prediction of the preferential tautomeric form adopted by compound (II) and other unsubstituted derivatives of this class of compounds in the solid state. The cohesion of the crystal is the result of van der Waals interactions and of one intermolecular N11—H11···O1^{i} hydrogen bond; N11···O1^{i} 2.882 (3), H11···O1^{i} 2.10 Å and N11—H11···O1^{i} 152° [symmetry code: (i) $1-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$].

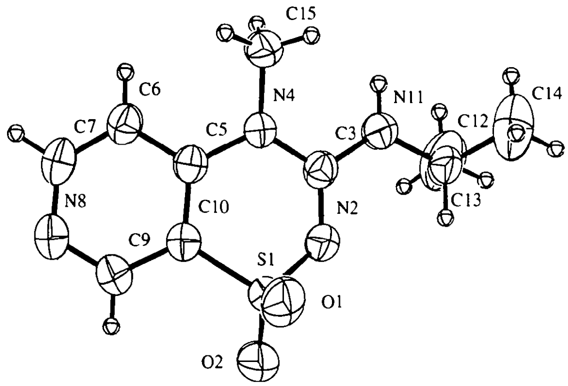


Fig. 1. The molecular structure of (I) with the atom-labelling scheme. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title compound was synthesized at the Laboratory of Medicinal Chemistry of Liège. The synthetic method has been published elsewhere (de Tullio *et al.*, 1996).

Crystal data

$C_{10}H_{14}N_4O_2S$
$M_r = 254.31$
Monoclinic
$P2_1/c$
$a = 6.2084\ (7)$ Å
$b = 13.0294\ (14)$ Å
$c = 15.0456\ (11)$ Å
$\beta = 95.709\ (6)°$
$V = 1211.0\ (2)$ Å^3
$Z = 4$
$D_x = 1.395$ Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å
Cell parameters from 24 reflections
$\theta = 35.76–40.90°$
$\mu = 2.371$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.53 × 0.38 × 0.19 mm
Colourless

Data collection

Stoe–Siemens AED four-circle diffractometer
ω-scans
Absorption correction: semi-empirical *via* ψ scan (*EMPIR*; Stoe & Cie, 1988*c*)
$T_{min} = 0.414$, $T_{max} = 0.574$
1658 measured reflections
1658 independent reflections
1283 observed reflections $[I > 2\sigma(I)]$
$\theta_{max} = 57.50°$
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 16$
2 standard reflections frequency: 60 min intensity decay: 3.8%

Refinement

Refinement on F^2
$R(F) = 0.0465$
$wR(F^2) = 0.1519$
$S = 1.072$
1658 reflections
158 parameters
H atoms restrained (included as riding atoms)
$w = 1/[\sigma^2(F_o^2) + (0.1063P)^2 + 0.2293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.363$ e Å^{-3}
$\Delta\rho_{min} = -0.238$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0167 (18)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.19458 (12)	0.61074 (6)	0.26042 (5)	0.0560 (4)
O1	0.3454 (4)	0.6676 (2)	0.2121 (2)	0.0751 (8)
O2	0.0746 (5)	0.6700 (2)	0.3183 (2)	0.0901 (9)
N2	0.3136 (4)	0.5204 (2)	0.3153 (2)	0.0544 (7)
C3	0.3713 (5)	0.4367 (2)	0.2737 (2)	0.0461 (7)
N4	0.2791 (4)	0.4061 (2)	0.1906 (2)	0.0446 (6)
C5	0.0862 (4)	0.4505 (2)	0.1531 (2)	0.0437 (7)
C6	−0.0501 (5)	0.4028 (2)	0.0862 (2)	0.0502 (8)
C7	−0.2328 (5)	0.4528 (3)	0.0513 (2)	0.0589 (9)
N8	−0.2993 (4)	0.5452 (2)	0.0788 (2)	0.0649 (8)
C9	−0.1732 (5)	0.5884 (3)	0.1442 (2)	0.0604 (9)
C10	0.0202 (4)	0.5465 (2)	0.1817 (2)	0.0492 (8)
N11	0.5239 (4)	0.3766 (2)	0.3149 (2)	0.0549 (7)
C12	0.6303 (5)	0.3991 (2)	0.4045 (2)	0.0595 (9)
C13	0.5249 (8)	0.3374 (4)	0.4717 (3)	0.101 (2)
C14	0.8698 (7)	0.3735 (5)	0.4085 (3)	0.110 (2)
C15	0.3644 (5)	0.3179 (2)	0.1451 (2)	0.0531 (8)

Table 2. *Geometric parameters (Å, °)*

S1—O2	1.427 (3)	C5—C6	1.394 (4)
S1—O1	1.445 (3)	C5—C10	1.397 (4)
S1—N2	1.579 (3)	C6—C7	1.367 (4)
S1—C10	1.738 (3)	C7—N8	1.352 (5)
N2—C3	1.326 (4)	N8—C9	1.321 (4)
C3—N11	1.333 (4)	C9—C10	1.387 (4)
C3—N4	1.381 (4)	N11—C12	1.471 (4)
N4—C5	1.397 (4)	C12—C13	1.492 (5)
N4—C15	1.464 (4)	C12—C14	1.519 (6)
O2—S1—O1	115.6 (2)	C6—C5—N4	122.7 (3)
O2—S1—N2	109.3 (2)	C10—C5—N4	120.8 (2)
O1—S1—N2	110.63 (15)	C7—C6—C5	119.1 (3)
O2—S1—C10	110.41 (15)	N8—C7—C6	125.0 (3)
O1—S1—C10	107.14 (15)	C9—N8—C7	115.6 (3)
N2—S1—C10	103.02 (14)	N8—C9—C10	124.0 (3)
C3—N2—S1	120.0 (2)	C9—C10—C5	119.7 (3)
N2—C3—N11	118.4 (3)	C9—C10—S1	122.9 (3)
N2—C3—N4	123.9 (3)	C5—C10—S1	117.3 (2)
N11—C3—N4	117.6 (3)	C3—N11—C12	122.9 (3)
C3—N4—C5	120.5 (2)	N11—C12—C13	109.0 (3)
C3—N4—C15	120.9 (2)	N11—C12—C14	109.9 (3)
C5—N4—C15	118.1 (2)	C13—C12—C14	110.4 (4)
C6—C5—C10	116.4 (3)		
O2—S1—N2—C3	156.7 (3)	C15—N4—C5—C10	−166.4 (3)
O1—S1—N2—C3	−74.9 (3)	N8—C9—C10—S1	173.9 (3)
C10—S1—N2—C3	39.3 (3)	C6—C5—C10—S1	−176.5 (2)
S1—N2—C3—N11	160.3 (2)	N4—C5—C10—S1	3.6 (4)
S1—N2—C3—N4	−21.4 (4)	O2—S1—C10—C5	−147.0 (2)
N2—C3—N4—C5	−13.7 (4)	O1—S1—C10—C5	86.3 (3)
N11—C3—N4—C5	164.7 (3)	N2—S1—C10—C5	−30.5 (3)
N2—C3—N4—C15	174.4 (3)	N2—C3—N11—C12	0.2 (4)
N11—C3—N4—C15	−7.3 (4)	N4—C3—N11—C12	−178.3 (3)
C3—N4—C5—C6	−158.5 (3)	C3—N11—C12—C13	98.3 (4)
C15—N4—C5—C6	13.7 (4)	C3—N11—C12—C14	−140.6 (4)
C3—N4—C5—C10	21.5 (4)		

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

The authors thank M. M. Vermeire for his helpful assistance in the diffractometry measurements and the Belgian FNRS (Fonds National de la Recherche Scientifique) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: PA1225). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bandoli, G. & Nicolini, M. (1977). *J. Cryst. Mol. Struct.* **7**, 229–240.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Pirotte, B., de Tullio, P., Lebrun, P., Antoine, M.-H., Fontaine, J., Masereel, B., Schynts, M., Dupont, L., Herchuelz, A. & Delarge, J. (1993). *J. Med. Chem.* **36**, 3211–3213.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Stoe & Cie (1988*a*). *DIF4. Diffractometer Control Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*b*). *REDU4. Data Reduction Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*c*). *EMPIR. Empirical Absorption Correction Program.* Version 1.2. Stoe & Cie, Darmstadt, Germany.
Tullio, P. de, Pirotte, B., Lebrun, P., Fontaine, J., Dupont, L., Antoine, M.-H., Ouedraogo, R., Khelili, S., Maggetto, C., Masereel, B., Diouf, O., Podona, T. & Delarge, J. (1996). *J. Med. Chem.* **39**, 937-948.

Acta Cryst. (1996). C**52**, 1743–1745

(5*R*)-5-[(*S*)-α-Hydroxybenzyl]-5-(L-menthyloxy)-4-(1-pyrrolidinyl)furan-2(5*H*)-one

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(Received 5 June 1995; accepted 22 January 1996)

Abstract

The determination of the structure of the title compound, $C_{25}H_{35}NO_4$, confirms the *R* and *S* absolute configurations at the 5 position of the furanone ring and the α position of the hydroxybenzyl substituent, respectively, taking into account the known configuration of the menthyl moiety.

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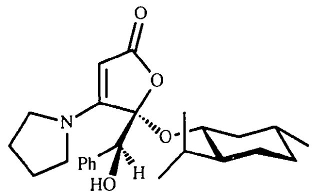
Comment

Due to their versatility as intermediates in organic synthesis, furan-2(5*H*)-ones have been the target compounds of several generally applicable synthetic routes. One of the most useful approaches is the reaction of enolate anions generated from furanone derivatives with electrophiles. In this case, the presence of an electron-releasing group such as alkoxy (Honda, Hayakawa, Kondoh, Okuyama & Tsubuki, 1991; Pelter, Al-Bayati, Ayoub, Lewis & Pardasani, 1987) or alkylamino (de Ancos, Fariña, Maestro, Martín & Vicioso, 1991; Nishide, Aramata, Kamanaka & Node, 1993; Schlessinger, Iwanowicz & Springer, 1988; Schlessinger, Mjalli, Adams, Springer & Hoogsteen, 1992) at the 4 position favours the formation of C5-substituted derivatives. With respect to the stereoselectivity of this reaction, several studies were carried out using substrates with a pyrrolidine derivative as chiral auxiliary group at the C4 position. Recently, a new 4-enaminofuranone with the chiral group at the 5 position of the furanone ring, 5-(L-menthyloxy)-4-(1-pyrrolidinyl)furan-2(5*H*)-one, has been prepared (Martín & Mateo, 1994) and the reaction of the enolate anion of this compound with several electrophiles has been studied (Martín & Mateo, 1995). The determination of the absolute configuration of the main product obtained in the reaction with benzaldehyde, the title compound, (I), has been accomplished by X-ray analysis and the known configuration of the menthyl moiety in the starting material.



(I)

The furanone ring can be considered to be flat, with the mean deviation of the plane for C4 [0.003 (5) Å]. The pyrrolidine ring has an envelope conformation with

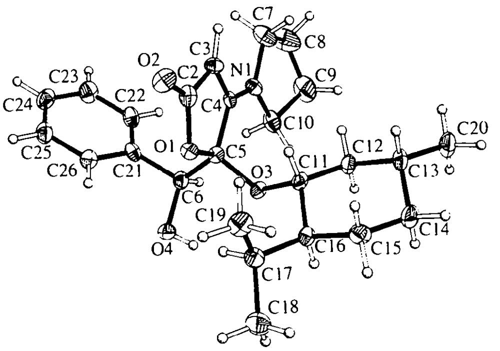


Fig. 1. The molecular structure of the title compound showing 20% probability displacement ellipsoids for non-H atoms.

ISSN 0108-2701